



High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR: accelerated identification of bioactive constituents in food and medicinal plants

Stærk, Dan

Publication date:
2015

Citation for published version (APA):
Stærk, D. (2015). *High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR: accelerated identification of bioactive constituents in food and medicinal plants*. Abstract from 2nd International Symposium on Profiling, Caparica - Lisbon, Portugal.

High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR: accelerated identification of bioactive constituents in food and medicinal plants

D. Staerk¹

1. Bioanalytical Chemistry and Metabolomics research group. Department of Drug Design and Pharmacology. Faculty of Health and Medical Science. University of Copenhagen. Universitetsparken 2, DK-2100 Copenhagen. Denmark.

Abstract

Purpose: Foods and medicinal plants are rich sources of bioactive constituents, but identification of these using bioactivity-guided fractionation is a time-consuming and laborious task. The purpose of our research is to advance profiling of bioactive constituents in foods and medicinal plants by analytical-scale microfractionation in 96-well plates followed by bioassaying, *i.e.*, microplate-based high-resolution bioactivity profiling [1,2].

Experimental description: Crude methanol or ethyl acetate extracts of selected food sources and medicinal plants were assessed for α -glucosidase-, α -amylase-, and aldose reductase inhibitory activity as well as radical scavenging activity. Extracts with inhibitory activities below 20 μ g/ml were subjected to microplate-based high-resolution bioactivity profiling for targeting subsequent HPLC-HRMS-SPE-NMR analyses towards bioactive constituents only. The experimental workflow is shown in **Figure 1**, and can be divided into *i*: analytical-scale HPLC separation, *ii*: micro-fractionation into 96-well microplates followed by aldose reductase inhibition assaying, α -glucosidase inhibition assaying, and ABTS^{•+} reduction assaying, *iii*: results from bioassays plotted against their respective retention time to produce triple high-resolution biochromato-gram, *iv*: identification of bioactive analytes from biochromatogram, *v*: HPLC-HRMS-SPE-NMR analysis targeted bioactive constituents, and *vi*: structural identification of bioactive constituents.

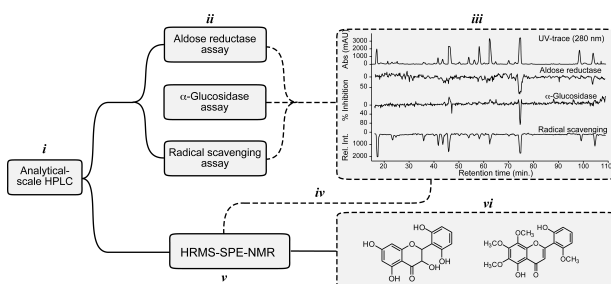


Figure 1. Experimental workflow for high-resolution profiling combined with HPLC-HRMS-SPE-NMR analysis

Results: High-resolution profiling of foods (seaweed, vegetables, spices, etc) and medicinal plants (e.g., traditional Chinese medicine) followed by structural characterization using HPLC-HRMS-SPE-NMR will be presented. This allowed identification of, *e.g.*, flavonoids, flavonoid glycosides, stilbenoids, stilbenoid glycosides, unsaturated fatty acids, *N-p*-comaroyloctopamine, and *N-p*-feruloyltyramine as antidiabetic principles in the investigated species [1-3].

Conclusions: High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR is an efficient technique for identification of known as well as new compounds direct from crude extracts of foods and medicinal plants.

Key Words: High-resolution bioassay, HPLC-HRMS-SPE-NMR, antidiabetic, functional food, medicinal plant.

Acknowledgements: Carlsbergfondet and the Danish Agency of Science, Technology and Innovation are acknowledged for financial support.

Correspondence: Professor Dan Staerk, Department of Drug Design and Pharmacology, University of Copenhagen, Universitetsparken 2, DK-2100 Copenhagen. E-mail: ds@sund.ku.dk.

[1] Liu, B.; Kongstad, K.T.; Qinglei, S.; Nyberg, N.T.; Jäger, A.K.; Staerk, D. *J. Nat. Prod.* **2015**, *78*, 294-300. [2] Schmidt, J.S.; Nyberg, N.T.; Staerk, D. *Food Chem.* **2014**, *161*, 192-198. [3] Kongstad, K.T.; Özdemir, C.; Barzak, A.; Wubshet, S.G.; Staerk, D. *J. Agric. Food Chem.* **2015**, *63*, 2257-2263.